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IS : 2080 - 1980
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Indian Standard
SPECIFICATION FOR STABILIZED HYDROGEN PEROXIDE
(First Revision)

First Reprint AUGUST 2000

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 ***February* 1981**

AMENDMENT NO. 1 JULY 1994
TO
IS 2080 : 1980 SPECIFICATION FOR STABILIZED HYDROGEN PEROXIDE

(*First Revision*)

(*Page* 8, *Appendix* B) — Add the following new clause before clause **B-1**:

'B-0 WARNING

It is essential that hydrogen peroxide be handled and stored so as to avoid the risk of violent decomposition or explosion. In particular only clean equipment compatible with the product should be used (for example, glass or unplasticized polyvinyl chloride) and containers should be vented to prevent build-up of pressure. Contact with all combustible substances should be avoided.

It is essential that personnel handling hydrogen peroxide be familiar with the detailed advice contained in the supplier's material safety data sheet, and be provided with protection against splashes in the eye and on the skin.

It is essential that eye protection be worn during the operation of this method.'

(CHD 003)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR STABILISED HYDROGEN PEROXIDE

(First Revision)

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(*Continued on page* 2)

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Indian Standard

SPECIFICATION FOR STABILIZED HYDROGEN PEROXIDE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 24 July 1980, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Hydrogen peroxide is mainly used as an oxidizing and bleaching agent in industry and also as disinfectant.

0.3 This standard was originally issued in 1962. Subsequently latest manufacturing technology has been introduced in the industry. Therefore, impurities prescribed in the earlier version of this standard (sulphates, total sulphates and phosphates, chlorides, nitrates and aluminium) have ceased to be relevant. Consequently, requirements for these characteristics have been deleted and new requirements have been added for residue on ignition, lead and arsenic.

0.3.1 Over the years the pattern of use has undergone a change and hydrogen peroxide is now increasingly used by other industries, namely, chemicals, electronics, paper, cosmetics, etc. These industries may have additional requirements for which a provision has been made in **3.5** of this standards.

0.4 Cosmetics and Toilet Goods Sectional Committee of ISI had recommended inclusion of another grade of nominal strength 3 percent, and incorporating the requirements of arsenic and lead for all the three grades. Additional requirement of non-volatile matter (residue on ignition) has been incorporated. This would limit metallic and inorganic impurities. Modifications have been made in the procedure for determination of strength eliminating the density chart.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for stabilized hydrogen peroxide.

2. GRADES

2.1 The material shall be of four grades as follows:

Grade 1 — of nominal strength 50.0 percent by mass,
Grade 2 — of nominal strength 35.0 percent by mass,
Grade 3 — of nominal strength 6.0 percent by mass, and
Grade 4 — of nominal strength 3.0 percent by mass.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of clear, colourless liquid, free from suspended impurities and dust particles.

3.2 Strength — The strength of the material, when tested according to the method prescribed in Appendix A, shall on the date of packing, be not less than 50.0 percent (*m/m*) for Grade 1, 35.0 percent (*m/m*) for Grade 2, 6.0 percent (*m/m*) for Grade 3 and 3.0 percent (*m/m*) for Grade 4.

3.2.1 This requirement shall also be met when the material has been stored and handled as prescribed in **4.1.**

3.3 Stability

3.3.1 *For Grades 1 and 2* — The material, when tested as prescribed in Appendix B, shall not evolve more than 0.3 ml of oxygen per minute per 25 ml at 100°C.

3.3.2 *For Grades 3 and 4* — The material when tested as prescribed in Appendix B, shall not lose its strength by more than 4 percent by mass of its original strength.

3.4 The material shall also comply with the requirements given in Table 1.

3.5 Additional Requirements — Additional requirements for specific uses, if required, may be prescribed as agreed to between the purchaser and the supplier (*see* **0.3.1**).

4. PACKING AND MARKING

4.1 Packing

4.1.1 All containers in which the material is packed shall be dry and clean.

TABLE 1 REQUIREMENTS FOR STABILIZED HYDROGEN PEROXIDE

(*Clause* 3.4)

SL No.	CHARACTERISTIC	REQUIREMENT			METHOD OF TEST (REF TO CL NO. IN APPENDIX C)
		Grade 1	Grade 2	Grades 3 & 4	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Acidity (as H_2SO_4), g/100 ml	0.015 to 0.030	0.015 to 0.030	0.012 to 0.025	C-1
ii)	Residue on evaporation. g/100 ml, *Max*	0.2	0.15	0.15	C-2
iii)	Residue on ignition, g/100 ml; *Max*	0.05	0.05	0.02	C-3
iv)	Iron (at Fe), g/100 ml, *Max*	0.000 1	0.000 1	0.000 1	C-4
v)	Copper (as Cu), g/100 ml, *Max*	0.000 01	0.000 01	0.000 01	C-5
vi)	Lead (as Pb), parts per million, *Max*	10	10	10	C-6
vii)	Arsenic (as As_2O_3), parts per million, *Max*	2	2	2	C-7

4.1.2 The material shall be packed in bottles or carboys as agreed to between the purchaser and the supplier. The material of construction of the container shall be compatible with the properties of hydrogen peroxide. Where the material of construction is glass, the bottles or carboys shall be made of neutral glass.

4.1.2.1 Containers may be of valve or vent type or without it. For containers with valve or vent arrangement, the minimum ullage should be 10 percent. For containers without valve or vent the ullage space shall be not less than one half of the total capacity of the container for Grades 1 and 2 and not less than one-fourth of the total capacity of the container for Grades 3 and 4. For Grades 1 and 2 the maximum capacity of the containers should be 2 litres, when hermetically sealed.

4.1.3 In the packaging of glass containers, suitable cushioning material shall be used.

4.1.4 All the containers shall be securely closed.

4.1.5 The containers shall be stored away from direct sunlight, preferably in the dark.

4.1.6 The containers shall be stored and handled as declared by the manufacturer to ensure stability.

4.2 Marking — The containers shall bear legibly and indelibly the following information:

a) Name and grade of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Mass of the material in kg;
d) Month and year of packing; and
e) Batch number.

4.2.1 Containers shall also prominently display the words 'CORROSIVE, HANDLE WITH CARE' and the appropriate symbol for labelling of dangerous goods prescribed in IS : 1260 (Part I)-1973*.

4.2.2 The product may also be marked with Standard mark.

4.2.3 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturer or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix D.

6. TEST METHODS

6.1 Tests shall be conducted as given in Appendices A, B and C. Reference to relevant appendices/clauses of appendices is given in **3.2, 3.3** and col 6 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Fictorial marking for handling and labelling of goods: Part I Dangerous goods (*first revision).*

†Specification for water for general laboratory use (*second revision).*

APPENDIX A

(*Clause* 3.2)

DETERMINATION OF STRENGTH

A-1. REAGENTS

A-1.1 Dilute Sulphuric Acid — 10 percent (*v/v*).

A-1.2 Standard Potassium Permanganate Solution — 1 N and 0.1 N.

A-2. PROCEDURE

A-2.1 For Grades 1 and 2

A-2.1.1 Weigh accurately about 1 g of the material in a 5 ml capacity weighing bottle. Measure about 100 ml of dilute sulphuric acid into a 500-ml conical beaker. Add potassium permanganate solution (0.1 N) dropwise until mixture assumes a faint permanent pink colour. Transfer the weighing bottle with the material carefully to the beaker and titrate with potassium permanganate solution (1 N) to the appearance of the same faint permanent pink colour obtained before the addition of the sample.

A-2.2 For Grades 3 and 4

A-2.2.1 Weigh accurately about 1 g of the material into a 5 ml capacity weighing bottle. Measure about 100 ml of dilute sulphuric acid into a 500-ml conical beaker. Add potassium permanganate solution (0.1 N) dropwise until the mixture assumes a faint pink colour. Transfer the weighing bottle with the material carefully to the beaker and titrate with potassium permanganate solution (0.1 N) to the appearance of the same faint permanent pink colour obtained before the addition of the material.

A-3. CALCULATION

$$\text{Hydrogen peroxide, percent by mass} = \frac{1{\cdot}7008 \times V N}{M}$$

where

V = volume in ml of standard potassium permanganate solution required for the titration,

N = normality of standard potassium permanganate solution, and

M = mass in g of the material taken for the test.

APPENDIX B

(*Clause* 3.3)

DETERMINATION OF STABILITY

B-1. FOR GRADES 1 AND 2

B-1.1 Apparatus

B-1.1.1 *Boiling Water-Bath*

B-1.1.2 *Reaction Tubes* — of size 150 × 25 mm with ground-glass cone.

B-1.1.3 *Liebig Condenser* – effective length 75 mm, ground-glass socket.

B-1.1.4 *Gas Burette* — with side arm and tap, 25 ml capacity, numbered in 1 ml division and graduation of 0.1 ml.

B-1.1.5 *Levelling Tube* — a little longer than the gas burette.

B-1.1.6 *Pipette* — with safety bulb, 25 ml capacity.

B-1.1.7 Complete assembly of the apparatus is shown in Fig. 1.

NOTE — All glass apparatus coming in contact with hydrogen peroxide should be:

a) of borosilicate glass,

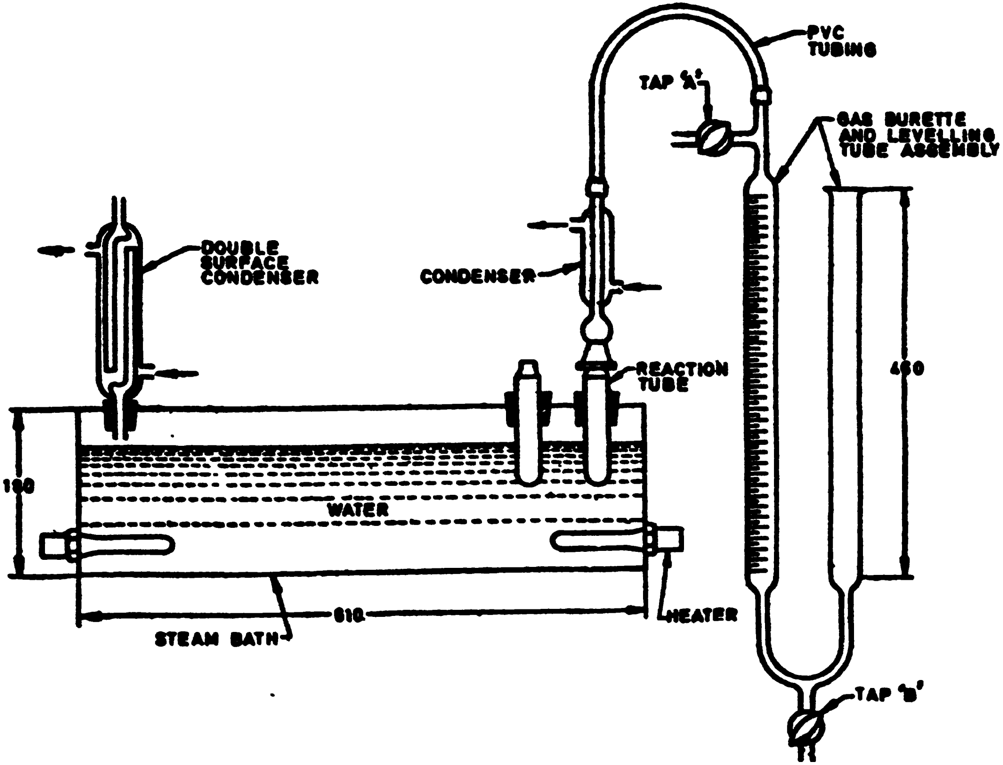
b) conditioned with nitric acid (relative density 1.42) at 100°C for 30 minutes and then well rinsed with water before its commissioning,

c) stored in dust free atmosphere, and

d) well rinsed with hydrogen peroxide before use.

B-1.2 Procedure — Pipette 25.0 ml of the sample into reaction tube and place the tube in continuously boiling and constant level water bath. Connect the condenser and open tap *A*. Allow 10 minutes for the condenser and reaction tube to equilibrate and adjust the water level in the gas burette to the zero mark by dropwise addition of water to the levelling tube. Close tap *A* and start the stop watch. Periodically equalize the water levels in the gas burette and the levelling tube by carefully and gradually draining water from tap *B*. Continue till maximum 20 ml of evolved oxygen is collected in the gas burette. Accurately equalize the water levels in the gas burette and the levelling tube in the manner described above and measure the volume of oxygen evolved. Let the duration of the test be *T* minutes.

NOTE — Separate reaction tubes should be used for each grade of hydrogen peroxide. Care should be taken never to allow the reaction tube to become completely dry during the course of an experiment.



All dimension as in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF STABILITY OF HYDROGEN PEROXIDE (GASOMETRIC METHOD)

B-1.3 Calculation

$$\text{Stability of the sample, ml of oxygen/min/25 ml} = \frac{V}{T}$$

where

V = volume in ml of oxygen evolved, and

T = duration of the test in minutes.

B-2. FOR GRADES 3 AND 4

B-2.1 Apparatus

B-2.1.1 *Round Bottom Flask* — of 250 ml capacity, fitted with a socket to fit in the reflux condenser (*see* **B-2.1.2**) and having a long neck. If new, the flask shall be pre-treated by boiling in it 100 ml of hydrogen peroxide (of the same strength as it is intended to test) under reflux for 30 minutes. It shall then be cleaned and kept exclusively for this test, Between tests, it shall be cleaned with nitric acid and rinsed thoroughly with water.

B-2.1.2 *Reflux Condenser* — Double surface, fitted with a cone to fit in the round-bottom flask (*see* **B-2.1.1**) and with an effective length of 15 to 20 cm.

B-2.1.3 *Preparation of Apparatus* — Wash the condenser with water, cover the top with a clean dust cap, namely, tube, beaker or other vessel of suitable size and allow to drain. Wash the flask with water and allow to drain. Draw about 50 ml of the material into a 100 ml pipette, rinse round the pipette and transfer to the round-bottom flask. Wash the flask and reject the washings.

B-2.2 Procedure

B-2.2.1 Pipette 100 ml of the material into the round-bottom flask and attach the condenser to the flask. Reflux gently on a sand-bath for exactly three hours, timing this period from the commencement of boiling. Cover the flask with a paper cap and cool rapidly under running water. *During all these operations, cars shall bo taken to exclude contamination with dust.* When the solution has cooled, determine its strength according to the method prescribed in Appendix A.

B-3. CALCULATION

B-3.1 Stability, percent loss in strength = $100 \times \frac{(A - B)}{A}$.

where

A = strength of the original material as obtained following the procedure prescribed in **A-2,** and

B = strength of the boiled material as obtained following the procedure prescribed in **B-2.2.1.**

APPENDIX C

(*Clause* 3.4)

METHODS OF TEST FOR STABILIZED HYDROGEN PEROXIDE

C-1. ACIDITY

C-1.1 Reagents

C-1.1.1 *Standard Sodium Hydroxide Solution* — 0.1 N.

C-1.1.2 *Rectified Spirit* — *See* IS : 323-1959*.

C-1.1.3 *Screened Methyl Red Indicator* — Weigh 0.050 g of methylene blue in a 100-ml beaker and dissolve in 50 ml of rectified spirit. Weigh 0.10 g of methyl red into a 100-ml beaker and dissolve in 50 ml of rectified spirit, by gently warming, if necessary. Mix the two solutions and store in an amber bottle. *The solution should not be used for more than three months.*

C-1.2 Procedure — Pipette out 100 ml of the material into a 400-ml beaker containing 100 ml of carbon dioxide-free water (freshly boiled), add a few drops of screened methyl red indicator and titrate with standard sodium hydroxide solution till the colour changes to green.

C-1.3 Calculation

$$\text{Acidity (as } H_2SO_4 \text{), g/100 ml} = 0.0490\ VN$$

where

V = volume in ml of standard sodium hydroxide solution, and

N = normality of standard sodium hydroxide solution.

C-2. DETERMINATION OF RESIDUE ON EVAPORATION

C-2.1 Procedure — Evaporate on a boiling water-bath 25 ml of the material to dryness in a weighed, dry platinum dish. Dry the residue to constant mass in an oven maintained at a temperature of 105 ± 2°C. Preserve the dish with contents for test in C-**3.**

C-2.2 Calculation

$$\text{Residue on evaporation, g/100 ml} = 4\ (\ B - A\ )$$

where

B = mass in g of the dish after drying; and

A = mass in g of the empty dish.

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*Specification for rectified spirit (*revised*).

C-3. DETERMINATION OF RESIDUE ON IGNITION

C-3.1 Procedure — Gently ignite the residue obtained in **C-2.1** in a furnace (800°C). Cool and weigh till constant mass is obtained.

C-3.2 Calculation

$$\text{Residue on ignition, g/100 ml} = 4 (B - A)$$

where

B = mass in g of the dish after ignition, and
A = mass in g of the empty dish.

C-4. IRON

C-4.1 Apparatus

C-4.1.1 *Nessler Cylinders* — 50 ml capacity (*see* IS : 4161-1967*).

C-4.2 Reagents

C-4.2.1 *Citric Acid Solution* — 10 percent (approx).

C-4.2.2 *Thioglycollic Acid*

C-4.2.3 *Ammonium Hydroxide* — 1 : 4 (*v/v*) (approx).

C-4.2.4 *Standard Iron Solution* — Dissolve 1.404 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in about 100 ml of water containing 10 ml of dilute sulphuric acid (10 percent *v/v*). Dilute to 1 000 ml. Take 50 ml of this solution in a 1 000 ml graduated flask and dilute again to mark. One millilitre of this diluted solution contains 0.01 mg of iron (as Fe). This diluted solution shall be freshly prepared.

C-4.3 Procedure

C-4.3.1 *Preparation of Solution* — Take 50 ml of the material into a beaker and add 5 ml of sodium hydroxide solution. Commence the decomposition by slight heating. When decomposition is complete and the material is free of active oxygen, cool. Dilute the cooled solution quantitatively to 100 ml.

C-4.3.2 Take 20 ml of the solution as prepared in **C-4.3.1.** Add 3 ml of citric acid solution and 5 drops of thioglycollic acid, mix and then add 10 ml of ammoniun hydroxide. Allow to stand for 5 minutes. Transfer the solution to a Nessler cylinder and dilute to 50 ml. Carry out a control test using 1 ml of standard iron solution carried out through the same steps as the test sample. Transfer to another Nessler cylinder and dilute to the same volume. Compare the intensity of colour produced in the two cylinders.

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*Specification for Nessler cylinders (*revised*).

C-4.3.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

C-5. COPPER

C-5.1 Apparatus

C-5.1.1 *Nessler Cylinders* — 50 ml capacity (*see* IS : 4161-1967*).

C-5.2 Reagents

C-5.2.1 *Sodium Hydroxide Solution* — approximately 0.5 N.

C-5.2.2 *Dilute Hydrochloric Acid* — approximately 0.5 N.

C-5.2.3 *Citric Acid Solution* — approximately 10 percent.

C-5.2.4 *Sodium Diethyldithiocarbamate Solution* — Dissolve 0.1 g of sodium diethyldithiocarbamate in 100 ml of copper-free water. Filter, if necessary. Preserve in an amber colour glass bottle and store in a dark place. This solution should not be used after two weeks.

C-5.2.5 *Ammonium Hydroxide* — approximately 1 : 4 (*v/v*).

C-5.2.6 *Standard Copper Solution* — Dissolve 0.392 8 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) in 1 000 ml of copper-free water. Pipette out 10 ml of this solution and dilute to 1 000 ml. One millilitre of the diluted solution contains 0.001 mg of copper (as Cu). The diluted solution should be freshly prepared before use.

NOTE — The purity of the salt should be checked before use.

C-5.2.7 *Extraction Solvent* — Two alternative extraction solvents, amyl alcohol and carbon tetrachloride, are prescribed. It is permitted to use any one of them.

C-5.3 Procedure — Take 100 ml of the material in a beaker and add 10 ml of sodium hydroxide solution. Heat gently until decomposition is complete and then add 10 ml of dilute hydrochloric acid. Heat and stir for about 5 minutes. Cool and add 2.5 ml of citric acid solution. Neutralize the excess acid with ammonium hydroxide and add 10 ml in excess, followed by 10 ml of sodium diethyldithiocarbamate solution. Transfer the solution to a separating funnel, stir and allow to stand for 5 minutes. Extract the yellow colour produced, with 10 ml of the extraction solvent. Repeat the extraction procedure further twice using 5 ml of the extraction solvent. Collect non-aqueous layers together, centrifuge to remove cloudiness. Alternatively, dry with anhydrous sodium sulphate. Make up the volume to 25 ml with fresh clear

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*Specification for Nessler cylinders (*revised*).

extracting solvent. Carry out a control test using 10 ml of standard copper solution, proceeding exactly as for the material under test. Compare the intensity of the colour produced in the two cylinders.

C-5.3.1 The limits laid down in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater that produced in the control test.

C-6. LEAD

C-6.1 Apparatus

C-6.1.1 *Nessler Cylinders* – 50 ml capacity (*see* IS : 4161-1967*).

C-6.2 Reagents

C-6.2.1 *Sodium Carbonate* — Solid.

C-6.2.2 *Dilute Nitric Acid* – 113 (*v/v*).

C-6.2.3 *Dilute Ammonium Hydroxide* — 1 : 2 (*v/v*).

C-6.2.4 *Standard Lead Solution* — Dissolve 1.598 g of lead nitrate (pre-dried for one hour at 100°C and cooled in a desiccator) in about 200 ml of water. Transfer it to a 1000-ml volumetric flask, add 50 ml of concentrated nitric acid and dilute to the mark with water. Mix well. Store in polyethylene bottle. One millilitre of this solution is equivalent to 1 000 µg of lead (Pb). This solution shall not be used for more than two weeks.

C-6.2.4.1 *Diluted standard lead solution* — Dilute 1.0 ml of standard lead solution (**C-6.2.4**) to 100 ml in a volumetric flask. One millilitre of this solution is equivalent to 10 µg of lead (Pb). The diluted solution shall be prepared afresh each time.

C-6.2.5 *Glacial Acetic Acid — See* IS : 695-1975†.

C-6.2.6 *Hydrogen Sulphide Gas or Sodium Sulphide Solution* — 1 percent (*m/m*).

C-6.3 Procedure

C-6.3.1 Evaporate to dryness in a suitable size silica dish 50 ml of hydrogen peroxide, 40 ml of water and 0.1 g of sodium carbonate on a steam or holing water-bath. Rinse the dish from sides towards centre with about 10 ml of water and again evaporate to dryness. Dissolve the residue in water and along with the washings make up the volume to 50 ml in a volumetric flask.

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*Specification for Nessler cylinders (*revised*).

†Specification for acetic acid (*second revision*).

C-6.3.2 Transfer 2 ml of the solution to a 50-ml Nessler cylinder and add 1 ml of dilute nitric acid and about 30 ml of water followed by 5 ml of dilute ammonium hydroxide. Make up the volume to 50 ml. Mix well.

C-6.3.3 Pipette out 2 ml of freshly prepared diluted standard lead solution into a second Nessler cylinder. Add 1 ml of glacial acetic acid, 30 ml of water and 10 ml of dilute ammonium hydroxide. Make up the volume to 50 ml with water. Mix well.

C-6.3.4 Pass hydrogen sulphide gas through the control and the sample solution at the rate of 2-3 bubbles per second, for 1 minute. Alternatively, 5 ml of sodium sulphide solution may be used in place of hydrogen sulphide gas. After 5 minutes compare the colour developed by the two solutions.

C-6.3.5 The limits prescribed in Table 1 shall be considered to have not been exceeded if the colour produced with the material is not greater than in the control test.

C-7. ARSENIC

C-7.1 Procedure — Take 5 ml of the sample solution (prepared as in **C-6.3.1**), and 15 ml of water and 10 ml of concentrated hydrochloric acid. Determine the arsenic by the modified Gutzeit method prescribed in **5.1** of IS : 2088-1971*, using for comparison a stain obtained with 0.01 mg of arsenic trioxide (as As_2O_3).

APPENDIX D

(*Clause* 5.1)

SAMPLING OF STABILIZED HYDROGEN PEROXIDE

D-1. SCALE OF SAMPLING

D-1.1 Lot — All the containers in a single consignment of the material of the same grade and drawn from a single batch of manufacture shall constitute a lot. If the consignment is found to consist of different batches of packing, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

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*Methods for determination of arsenic (*first revision*).

D-1.2 For ascertaining the conformity of the material in the lot to the requirements of the specification, samples shall be tested separately from each lot. The number of containers to be selected at random from the lot shall depend upon the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

No. OF CONTAINERS IN THE LOT (*N*) (1)	SAMPLE SIZE (*n*) (2)
Up to 50	5
51 „ 100	7
101 „ 300	10
301 „ 1 000	15
1 001 and above	20

D-1.2.1 In order to ensure randomness of selection, random number tables shall be used. For guidance and use of the random number tables reference may be made to IS : 4905-1968*. In case these tables are not available, the following procedure is recommended for use:

> Starting from any one container, count them as 1, 2,........ *r*, where *r* is the integral part of *N/n* (*N* and *n* being the lot size and sample size respectively). Every *r*th container thus counted shall be withdrawn to constitute the sample.

D-2. PREPARATION OF TEST SAMPLES

D-2.1 From each of the containers selected according to **D-1.2.1**, a representative portion of the material, sufficient to make triplicate determination for all the requirements given in 3, shall be drawn.

D-2.2 Out of these portions a small but approximately equal quantity of the material shall be taken and thoroughly mixed to form a composite sample, sufficient to carry out triplicate determination for all the characteristics tested on the composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

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*Methods for random sampling.

D-2.3 The remaining portions of the material from each container shall be divided into three equal parts each forming an individual sample, one set of individual samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

D-2.4 All the individual and composite samples shall be transferred to separate containers. These containers shall be sealed air-tight and labelled with full identification particulars.

D-2.5 The referee sample consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

D-3. NUMBER OF TESTS

D-3.1 Tests for the determination of strength (*see* **3.2**) and stability (*see* **3.3**) of the material shall be conducted on each of the individual test samples and the test results shall be recorded separately for different test samples.

D-3.2 Tests for the determination of all characteristics given in Table 1 shall be carried out on the composite test sample.

D-4. CRITERIA FOR CONFORMITY

D-4.1 From the results recorded according to **D-3.1** the mean ($\bar{X}$) and range (R) of test results for any characteristic shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of test results } 1, 2, \ldots\ldots\ldots n}{\text{Number of test results, } n}$$

Range (R) = Difference between the maximum and minimum of the test results.

D-4.2 In the case of samples of size 10 or more, every 5 test results shall be grouped together and the range of each group shall be determined. Average range ($\bar{R}$) shall then be computed from the group ranges at given below:

$$\text{Average range } (\bar{R}) = \frac{\text{Sum of group ranges}}{\text{Number of groups}}$$

D-4.3 The lot shall be declared as conforming to the requirements of the specification for different characteristics if the corresponding criteria for conformity given below are complied:

Characteristics	*Criteria for conformity*
a) Strength	($\bar{X} - 0.6R$*) shall be greater than or equal to the relevant limit prescribed in **3.2**
b) Stability (percent loss in strength)	($\bar{X} + 0.6R$*) shall be less than or equal to the relevant limit prescribed in **3.3**
c) Those listed in Table 1	Tests results on the composite test sample shall meet the corresponding requirement given in Table 1

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*When the sample size is 10 or more, a average range ($\bar{R}$) (*see* **D-4.2**) shall be used in place of range (R).

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